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Report on;

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for Research Entitled:

"Effects of High Energy Radiation on the Mechanical Properties of Epoxy/Graphite Fiber Reinforced Composites"

Covering the period January 1, 1984 - December 31, 1984

(NASA-CR-174314) EFFLCIS OF HIGH ENERGY N85-17047 RADIATION ON THE MECHANICAL PROPERTIES OF EPOXY-GRAPHITE FIBER KEINFCROED COMPOSITES Research Report, 1 Jan. - 31 Dec. 1984 Unclas (North Carolina State Univ.) 58 p G3/24 11502

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leveling of about 2-5 x 10^{19} radicals/g. Therefore, a graphite composite containing about 2/3 fiber mass would have its ESR signal dominated by the graphite. Indeed, as shown also in Figure 17, the signal of an unirradiated composite shows-essentially the same lineshape as that of fibers and the concentration is attributable to the fibers.

When fibers were irradiated (up to 5×10^3 Mrad of 0.5 Mev electrons), no measurable increase in radical concentration was observed. Further, when composites were irradiated, the ESR spectra remained totally dominated by the signal from the graphite fibers (the estimated maximum height of the signal attributed to the epoxy is less than 1% of the maximum signal of that of the fibers). Whether the radicals on the fibers play a role in the decay and reaction of radicals in the matrix has not yet been determined.

3. The Effect of Radiation Temperature on the ESR Lineshape

Earlier we reported that the ESR signal of TGDDM/DDS epoxy irradiated (γ^{60} Co) and maintained at liquid N₂ (77°K) is a broad line with even broader shoulders that disappear within minutes at room temperature. The line remaining after a few minutes at room temperature is approximately symmetric and appears to be a singlet.

Shown in Figure 18 are the spectrum (I + II) of an epoxy irradiated and maintained N_2 temperature, the spectrum (I) of the same sample after 30 minutes at room temperature, and the difference spectrum (II). Spectrum II shows that the signal of the short-lived radicals may be separated from that of the long-lived radicals and that the former signal appears to be either a sextet or an octet. Therefore, there may be as many as four different short-lived radicals present. We feel that the subtraction technique combined with measurements of lineshape changes with tempearture will provide valuable information about the species of

radicals generated by ionizing radiation and the reaction mechanisms following radiation.

A difference was observed in the lineshapes of samples that were irradiated in air at room temperature and samples that were irradiated in liquid nitrogen and brought to room temperature (in air) for various times. There is a clearly observable asymmetry in the lineshapes of samples irradiated with either γ or electron irradiation in air while samples irradiated at (60 Co γ) at 77°K then brought to room temperature appeared approximately symmetrical (see FIGURE 19). If the samples irradiated in air are left at room temperature for long times (Figure 20) the lineshape changes while the lineshape of the sample irradiated in liquid N₂ remains approximately constant. This suggests that oxygen diffuses into the samples irradiated in air, reacts with the radicals and is responsible for the relatively narrow line observed in Figure 20.

In an attempt to eliminate the effect of oxygen, a sample of epoxy was prevacuumed for seven days at room temperature, sealed in Al foil, and then it was irradiated with 60 Co * at room temperature. Again, the lineshape appeared to be essentially identical to the sample which was left in air and irradiated (see Figure 21). This suggests that elimination of oxygen was not achieved. Another explanation is that a different mechanism of radical decay occurs in samples irradiated at $^{+77}$ CK and warmed to room temperature after radiation.

4. Proposed Mechanism of Carbonyl Group Formation

There are two carbonyl peaks in the IR spectra of epoxy film after irradiation (Figure 15), vix, at 1720 cm⁻¹ and 1660 cm⁻¹. The peak at 1720 cm⁻¹ is attributed to ketones or carboxylic acids and the peak at 1660 cm⁻¹ is due to an amide group.

A possible mechanism for the carbonyl formation is as follows:

That is, carboxyl group formation may accompany back-bone scission. The radicals produced, can also lead to crosslinking.

The oxidation of the methylene bridge of TGDDM seems to be very minor since the carbonyl peak at 1720 cm⁻¹ is at a much higher wavenumber than the expected for the carbonyl

in I. Due to the highly conjugated structure of I, its carbonyl peak must appear at lower wavenumber than 1715 cm⁻¹ which is normal for a ketone.

I

Amide Formation

There are two possible sites for amide formation; DDS sites and TGDDM sites. The amide formation at the DDS site is less favorable than at TGDDM since the electron density at the nitrogen in DDS site is lower than that in TGDDM site.

Therefore the IR peak at 1660 cm⁻¹ is likely to be due to the amide formation in TGDDM. The increase of this peak was less than 15% with a 1,000 Mrad radiation dose.

VI. Summary Discussion

The three point flexural stress and flexural modulus and the interlaminar shear stress (either in compression or tensile) data generally increase with radiation doses up to 10,000 Mrad (ignoring what is apparently a sample to sample variation, probably due to fabrication variation). The increase is particularly striking for the ILS strengths. The room temperature elastic modulus (measured on the Rheovibron) of the epoxy itself is reduced 50% after a 3100 Mrad dose. This indicates that considerable chain scission occurs in the epoxy on irradiation with high energy radiation particularly in the presence of oxygen.

ESR spectra of irradiated epoxy samples show radical species with at least two characteristic half-lives, one short-lived and one long-lived. The short-lived species are probably in regions of low crosslink density and due to their relatively high mobility either re-combine or react with oxygen. Either mode of decay could lead to additional crosslinking. The long-lived radical species are probably in areas of relatively high crosslink density, have lower mobilities which reduces their rate of recombination or crosslinking and their rate of reaction with oxygen due to their lower accessibility.

The evidence from surface studies, both contact angle and ESCA measurements, and infrared spectroscopy demonstrates convincingly that the radicals produced by irradiation react with oxygen. There are two possibilities that are suggested: (1) In spite of the fact the samples are evacuated prior to irradiation there is probably some residual oxygen in the samples, plus oxygen may have leaked into the system during irradiation. (2) Even if oxygen removal prior to irradiation was complete and the system was tight, reaction of the chain radicals with oxygen would occur rapidly on exposure of the samples to air because of the abundance of long-live radical species.

chain scission and crosslinking occur concurrently when polymers are exposed to ionizing radiation. Usually one process dominates. The dynamic mechanical test suggests that chain scission dominates in the epoxy. The three-point bending tests and the ILS strength data indicate there is chain scission initially occurring in the epoxy matrix of the composite followed by crosslinking or interaction with the epoxy chain radicals with radicals on the surface of the graphite fibers, resulting in improved ILS strengths.

The ESR spectrum of unirradiated graphite fibers show there is an abundance of free radicals present (ca. $10^{20}/g$).

We have previously demonstrated that graphite fibers are not damaged by irradiation.

The problem that must be addressed is what would occur in a completely oxygen-free atmosphere, that is, in space? Undoubtedly, crosslinking would occur.

Of course, the degree of crosslinking would be dependent on the ability of the chain radicals to migrate over considerable distances. However, crosslinking at the epoxy-graphite fiber interface could probably occur quite readily leading to improved ILS strengths.

Chain scission could also occur as shown below:

but whether this disproportionation would occur with any ease is somewhat debatable. Certainly, chain scission would occur more readily in the presence of oxygen.

Though speculative in the absence of test data on specimens irradiated and tested in the complete absence of oxygen, it would appear that irradiation with high energy radiation will cause an increase in the mechanical properties of epoxy/graphite fiber composites in a space environment.

VII. Tables and Figures

Table I Summary of Mechanical Tests on Irradiated Composites

Composite	Fiber Arrangement	Maximum Radiation Dose	Test*	Trend**
T300/5208	Longitudinal	8,000 Mrad	ТРВ	I
T300/5208	Longitudinal	10,000 Mrad	ILS-TU	D
T300/5208	0/±45/0	10,000 Mrad	ТРВ	I
T300/5208	0/±45/0	10,000 Mrad	ILS-TU	D
T300/5208	90/±45/90	10,000 Mrad	ТРВ	I
T300/5208	90/±45/90	10,000 Mrad	ILS-TU	D
T300/5208	Transverse	10,000 Mrad	ТРВ	I
C6000/PMR15	Longitudinal	8,000 Mrad	ТРВ	I
C6000/PMR15	Transverse	10,000 Mrad	TPB	I
C6000/PMR15	Longitudinal	10,000 Mrad	ILS-TU	D
T300/5208	Longitudinal	9,000 Mrad	ILS-CS	I
T300/5208***	Longitudinal	9,000 Mrad	ILS-TU	Ì
T300/5208***	Longitudinal	9,000 Mrad	ILS-TS	I
T300/5209	Longitudinal	9,000 Mrad	ILS-CS	I
T300/5209***	Longitudinal	9,000 Mrad	ILS-TU	D
T300/5209***	Longitudinal	9,000 Mrad	ILS-TS	I
T300/5208	Longitudinal	10,000 Mrad	ILS-TU	I
T300/5208	0/±45/0	. 10,000 Mrad	ILS-TU	I
T300/5209	Longitudinal	7,500 Mrad	ILS-TU	I

^{*}ILS = Interlaminar shear **I = ILS. increames with radiation dose

D = ILS decreases with radiation dose ***Samples tested previously in ILS-CS mode

TU = Tensile, unsupported TS = Tensile, supported CS = Compressive, supported

Table II.

Interlaminar Shear Stress by Compressive Force
with a Side Support

Sample	Radiation Dose (Mrad)	No. of Specimens	Shear Stress (Kg/cm ²)	Standard <u>Deviation</u>	<u>%CV</u>	% change to control
T300/5203	0	3	581	87.8	15.1	0
longitudinal	3000	5	654	48.3	7.3	+12.6
	6000	5	667	94.5	14.1	+14.8
	9000	5	784	51.6	6.5	+34.9
T300/5209	0	3	701	68.8	9.8	0
longitudinal	3000	5	765	158.3	20.7	+9.1
(notched after	6000	5	843	92.0	10.9	+20.3
irradiation)	9000	5	909	148.9	16.4	+29.7
T300/5209	6000	2	881	29.0	3.3	+25.7
longitudinal	9000	3	778	67.7	8.7	+11.0
(notched before						

irradiation)

Table III.

Interlaminar Shear Stress by Tensile Force with a Side Support (samples loaded previously in ILS-CS mode)

Sample	Radiation Dose (Mrad)	No. of Specimens	Shear Stress (Kg/cm ²)	Standard Deviation	%CV	% change to control
T300/5208	0	2	181.8	8.1	4.4	0
longitudinal	3000	5	186.6	28.2	15.1	+2.6
	6000	4	173.9	20.1	11.6	-4.3
	9000	4	215.0	18.1	8,4	+18.3
T300/5209	0	3	189,6	17.6	9.3	0
longitudinal	3000	4	205.2	21.9	10.7	+8.2
	6000	5	251.7	31.0	12.3	+32.8
	9000	5	234.0	38.7	16.6	. +23.4

Table IV.

Interlaminar Shear Stress by Tensile Force without a Side Support (samples loaded previously in ILS-CS mode)

Sample	Radiation Dose (Mrad)	No. of Specimens	Shear Stress (Kg/cm ²)	Standard Deviation	%CV	% change to control
T300/5208	O	3	145.7	9,8	6.8	0
longitudinal	3000	5	133.7	25.5	19.1	-8.2
	6000	5	146.6	15.8	10.8	+0.6
	9000	5	174.5	18.1	10.3	+19.8
T300/5209	0	3	153.9	7.6	4.9	0
lõngitudinal	3000	5	171.9	25.3	14.7	+11.7
	6000	5	159.4	13,2	8.3	+0.4
	9000	5	123.1	17,3	14.1	_20.0

Table V.

Interlaminar Shear Stress by Tensile Force without a Side Support

Sample	Radiation Dose (MRAD)	No. of Specimens	Shear Stress (Kg/cm ²)	Standard Deviation	%CV	% change to control
T300/5208	0	9	154,1	20.8	13.5	0
longitudinal	2500	7	166.6	28.6	17.1	+8.1
	5000	10	176.2	28.0	15.9	+14.3
	7500	10	172.8	25.9	15.0	+12.1
	10000	8	157.1	16.3	10.3	+1.9
T300/5208	0	5	152,1	35.4	23.3	0
0/±45/0	2500	6	185.2	24.1	12.3	+21.8
	5000	9	193.0	27.2	14.1	+26.9
	7500	6	200.8	15.0	7.5	. +32.0
	10000	7	246.1	21.9	8.9	+61.8
T300/5209	0	10	183.1	31.8	17.4	0
longitudinal	2500	10	215.5	21.3	9.9	+17,7
	7500	10	254.4	32.8	12.9	+38.9

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Table VI. Surface Tension Properties of Test Liquids at $20^{\rm O}{\rm C}$

Test Liquid	<u>α</u> L	βL	YLdA	A L A	YLA
Water	4.67	7.14	21.8	51.0	72.8
Formamide	5.68	5,10	32.3	26.0	58.3
Ethylene- glycol	5.41	4.35	29.3	19.0	48.3
1-Bromo- naphthalene	6.68	0.00	44.6	0.0	44.6
Tricresyl- phospate	5.10	3.26	26.0	10.6	36.6
Hexadecane	5.25	0.00	27.6	0.0	27.6
Hexane	4.29	0.00	18.4	0.0	18.4

<u>Note</u>:

 α_{L}/β_{L} ; $(dyne/cm)^{1/2}$

*LA; dyne/cm

Table VII.
Surface Energy of Irradiated Epoxy Films

Dose Mrad	X EV	XPEA -dyne/cm	8 _{EA}	8 EA/8EA
0	22.9	1.2	24.1	0.05
400	23.3	11.3	34.6	0,33
1,000	21.0	39.7	60.7	0.65
2,000	22.6	38.0	60.5	0.63
€,000	22.7	38.2	60.8	0.63
10,000	21.9	43.3	65.2	0,66

Table VIII.
Surface Energy of Irradiated Graphite Fibers

Dose Mrad	X d GA	y GA _dyne/cm	GA GA	8 GA/8GA
0	21.2	34.5	55.7	0.62
1,000	20.1	42.3	62.3	0.68
5,000	20.0	42.6	62.6	0.68
10,000	19.3	44.6	64.0	0.70

Table IX.
Relative Atomic Concentration of C and C

<u>Sample</u>	•- <u>c</u>	0	<pre>0/C(irradiated) 0/C(control)</pre>
Epoxy Film			
As cured	1	0.237*	1.37
10,000 Mrad	1	0.324*	1.37
Composite (fracture surface)			
Control	1	0.237	1.50
10,000 Mrad	1	0.358	

^{*}Average value of two measurements for the same sample.

. Table X.

Contact Angle of Composite Fracture Surfaces (degree)

	<u>Control</u>	7,500 Mrad
Fiber direction*	••	
Advancing	61.7	45.2
Receding	-	•
Lateral direction*		
Advancing	85	59
Receding	34	59
Outermost surface		
Advancing	. 88	54.3
Receding	83.3	53.6

^{*}Shear-fractured surface

Note:

Sample; T300/5209, Uniaxial

Liquid; H₂0

T300/5208

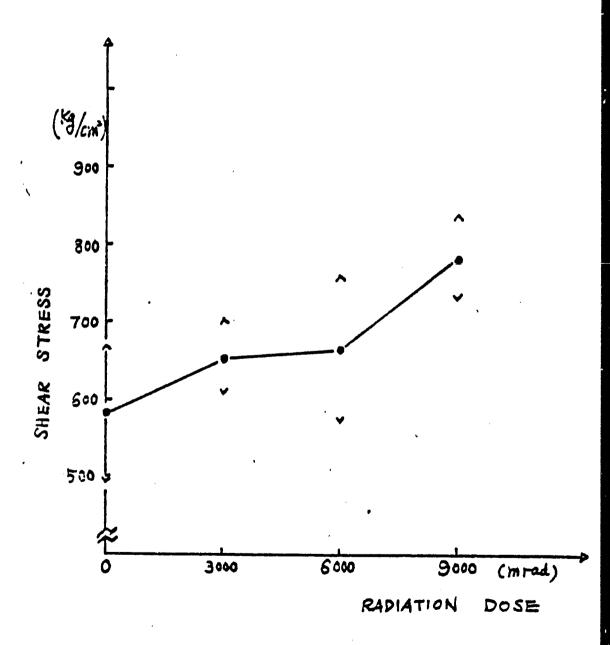


Fig. 1. Interlaminar Shear Stress versus radiation dose by compression using supporting fixture.

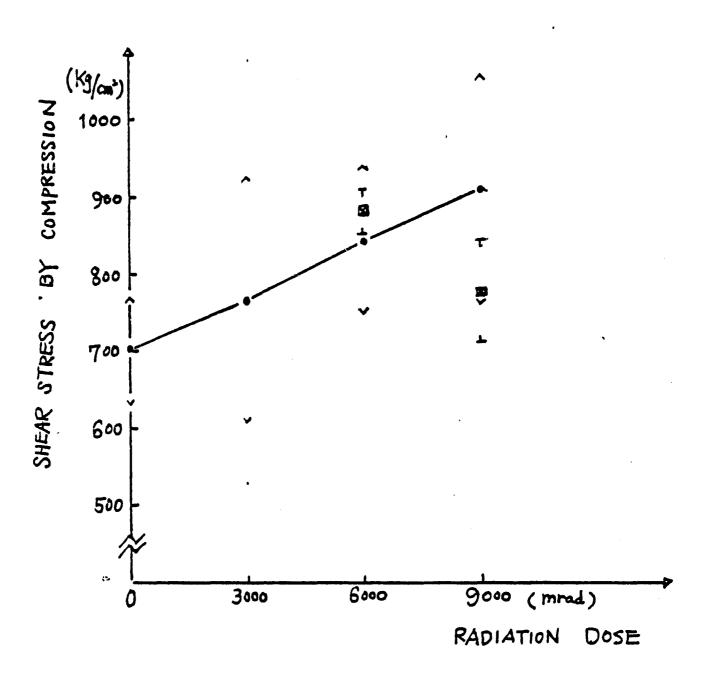


Fig. 2. Interlaminar Shear Stress versus radiation dose by compression using supporting fixture.

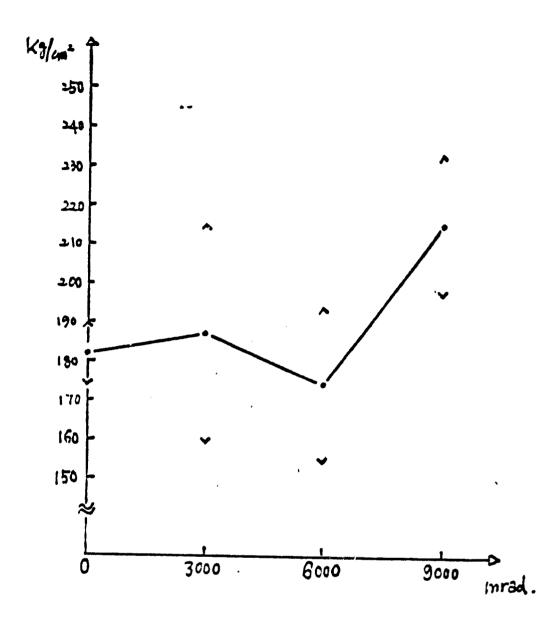
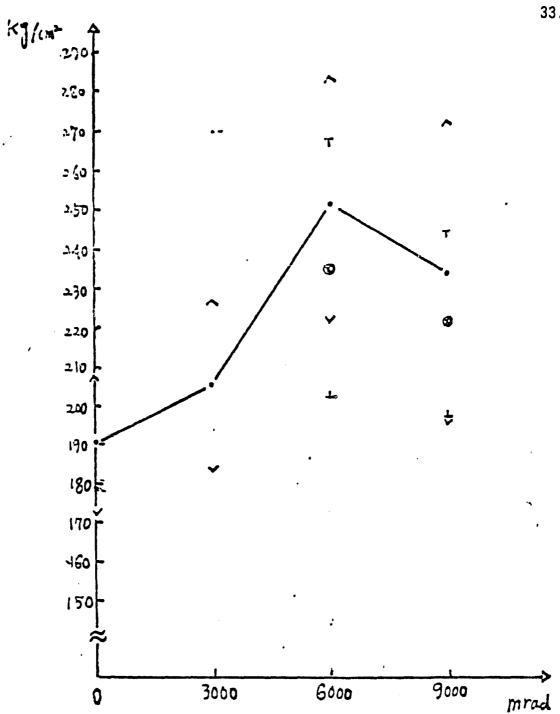
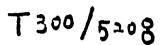


Fig. 3. Interlaminar shear stress versus Radiation dose in tension mode using supporting plate.





Interlaminar Shear Stress versus Radiation dose in tension mode using supporting plate.



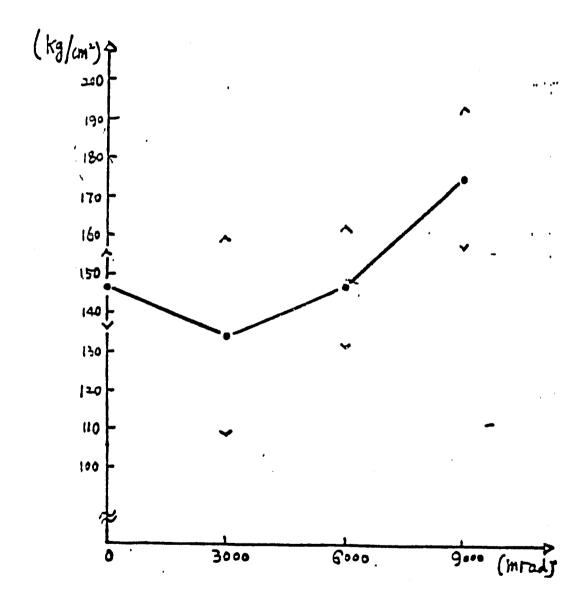


Fig. 5. Interlaminar Shear Stress versus Radiation dose in tension mode without using supporting plate.

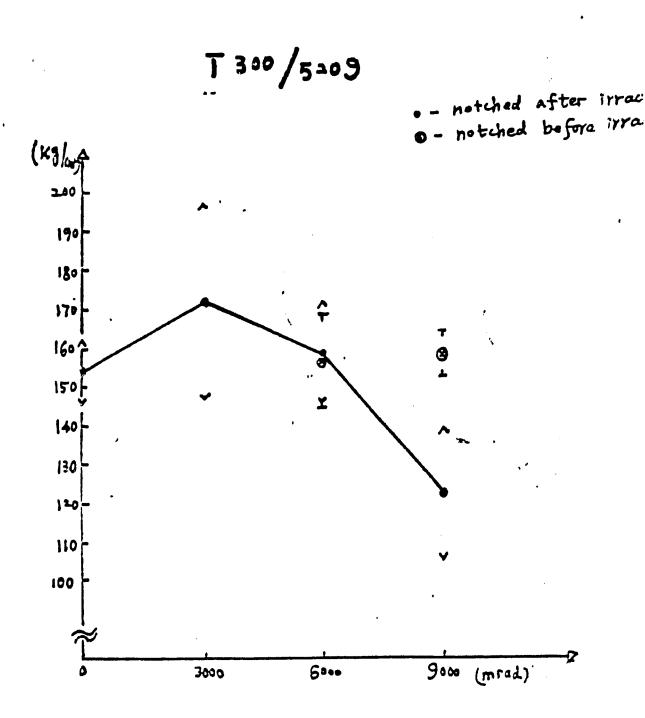


Fig. 6. Shear Stress versus Radiation dose in tension mode without using supporting plate.

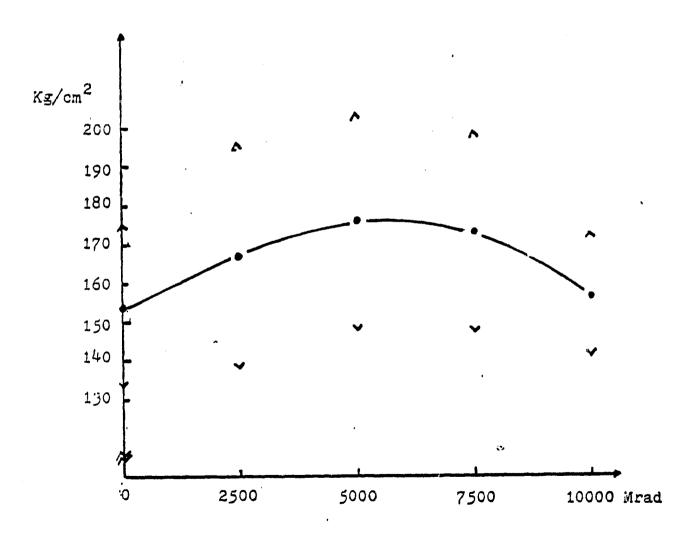


Fig. 7. Interlaminar Shear Stress in Tension mode without a supporting fixture for T300/5208 longitudinal samples.

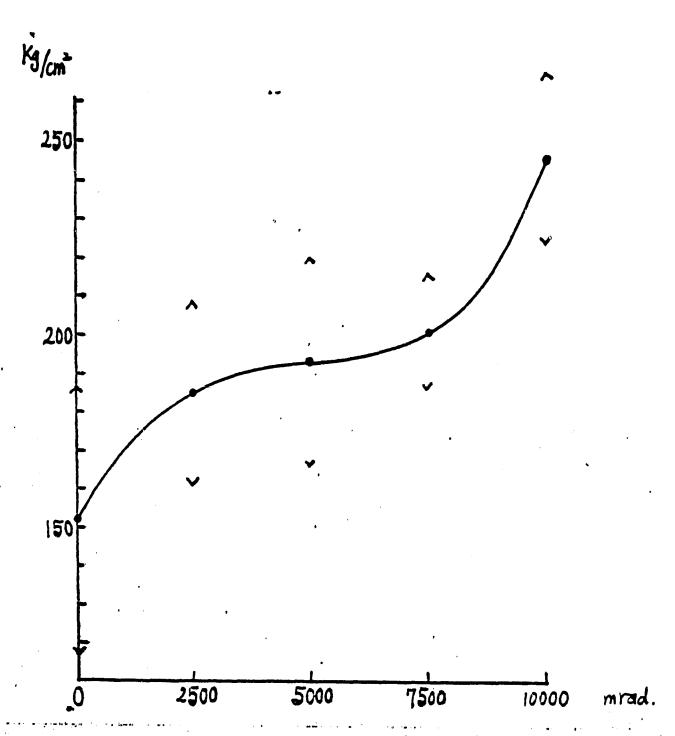


Fig. 8. I.L.S.S. in Tension mode w/o supporting fixture for T300/5208 0/± 45/0 pTy.

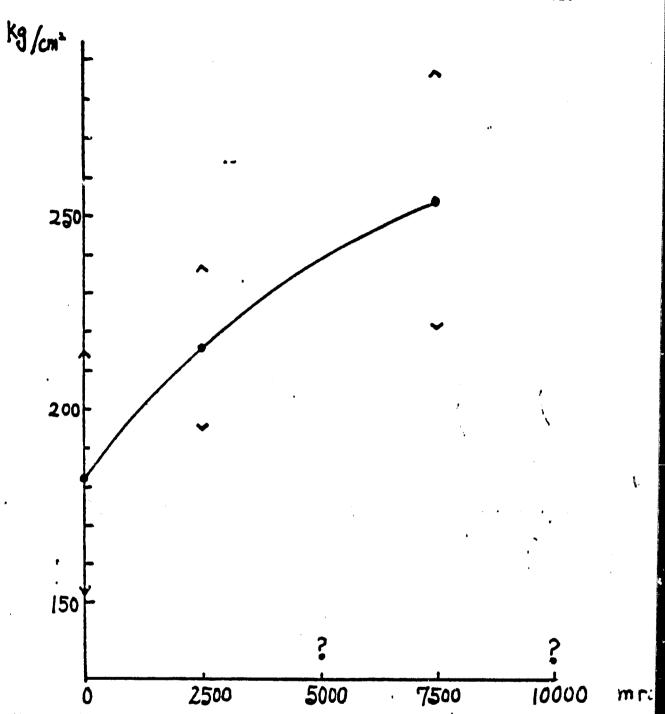


Fig. 9. I.L.S.S. in Tension Mode w/o supporting fixture for T300/5209 Uni-Dir.

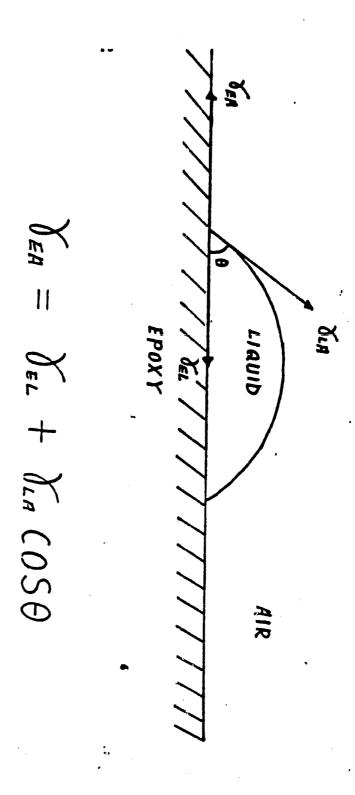
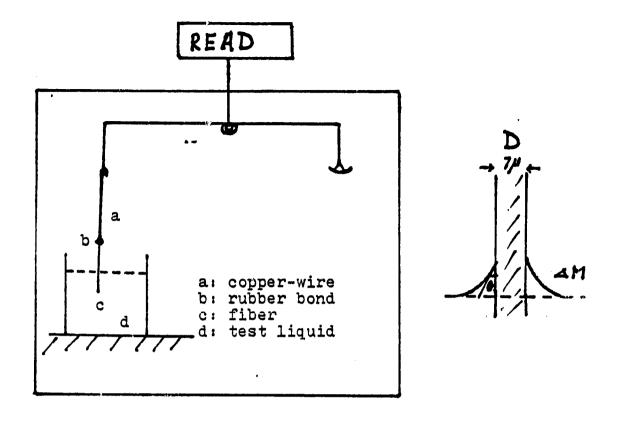


FIG. 10 FORCES ACTING ON A LIQUID DROP RESTING ON THE EPOXY FILM.



$$\Delta M \cdot g = \pi D_{\chi} \chi_{LA} \cos \theta$$

$$\cos\theta = \frac{\Delta M \cdot 9}{\pi D \chi_{A}}$$

Fig. 11. A schemati; diagram of Wilhelmy technique.

 ΔM : weight difference before and after immersion of the fiber, g : 980.6 dyne/cm, D : diameter of the fiber, \textbf{Y}_{LA} : surface tension of the test liquid.

EPOXY (TGDDM-DDS)

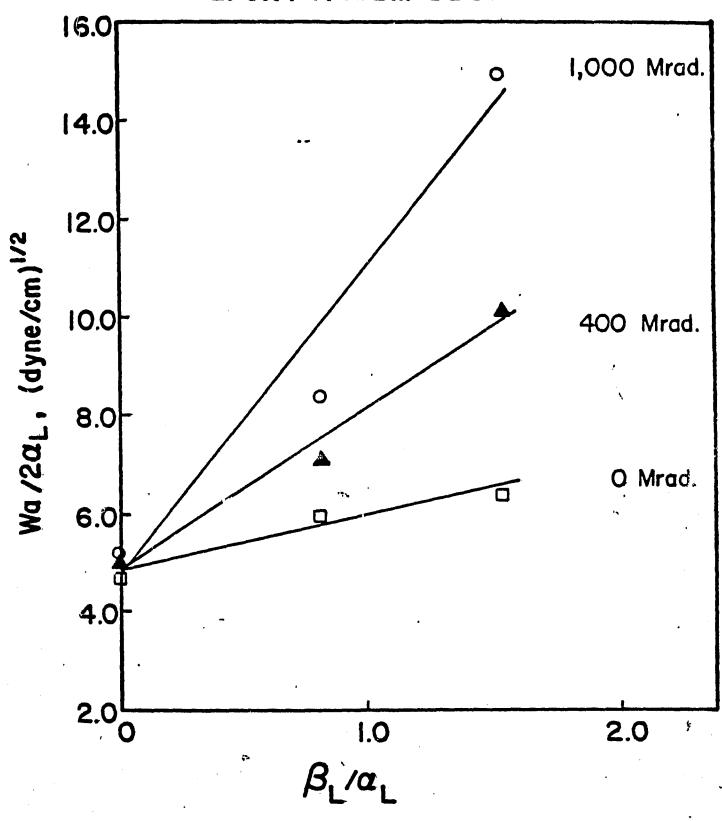


Figure 12. Plot of $W_a/2\alpha_L$ against β_L/α_L of Epoxy.

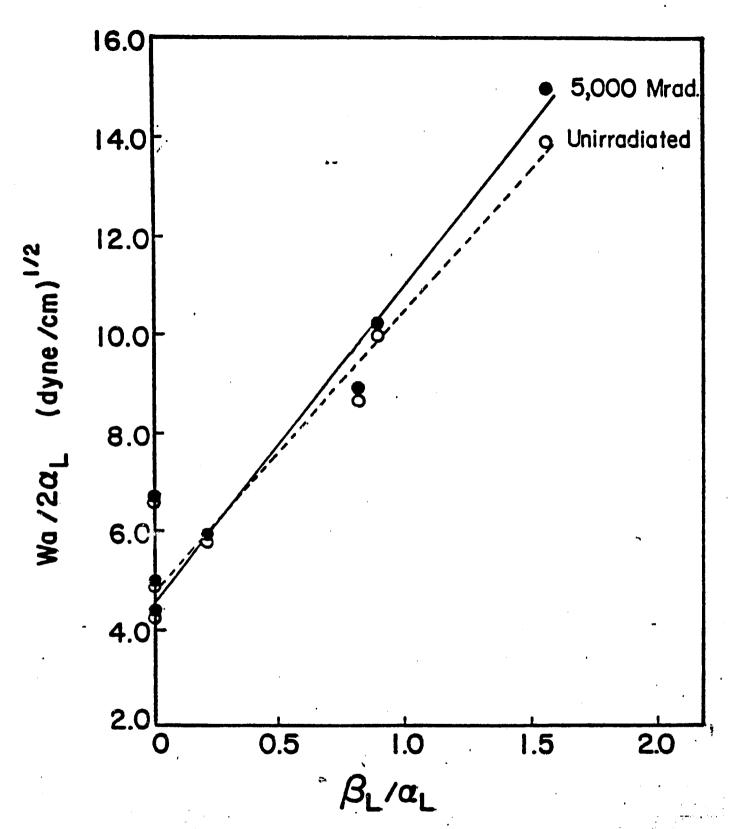
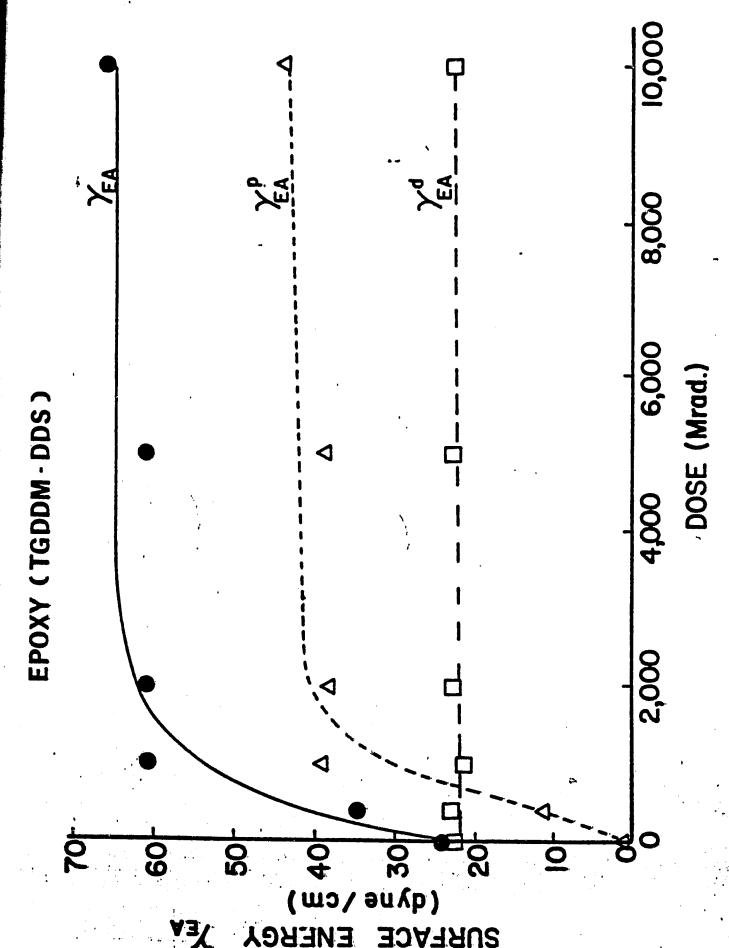
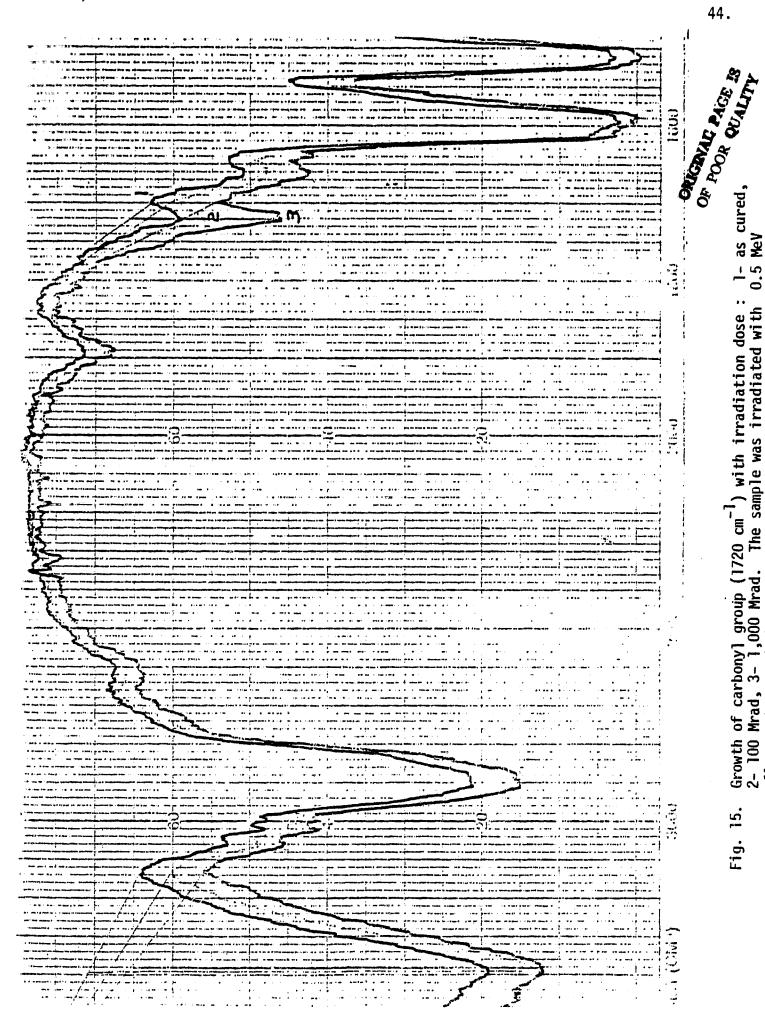


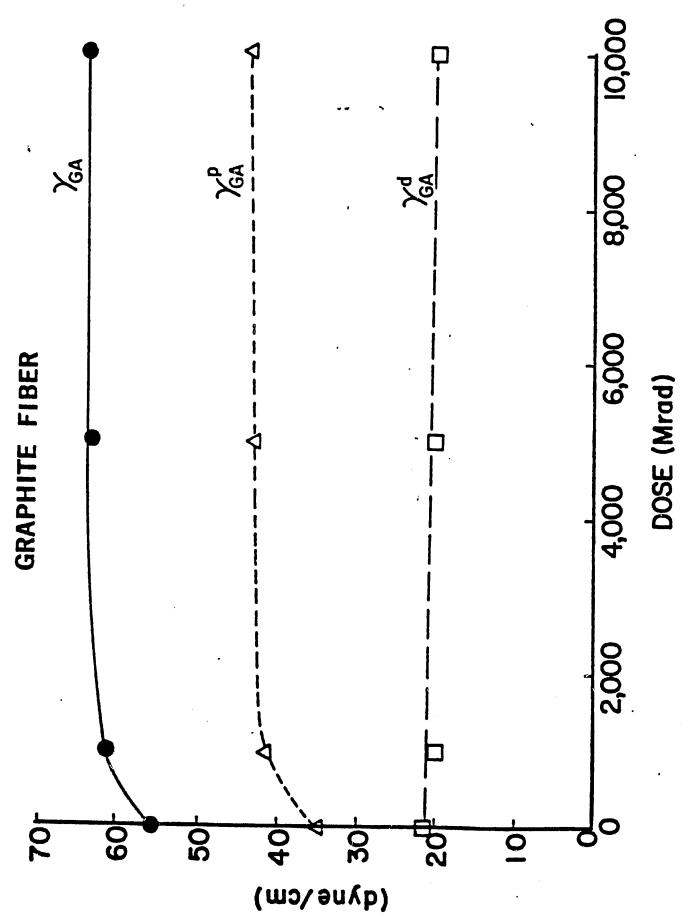
Figure 13. Plot of $\sqrt[4]{20}$ against $\sqrt[3]{2}$ of graphite fiber.



Plot of surface energy against irradiation dose of epoxy.



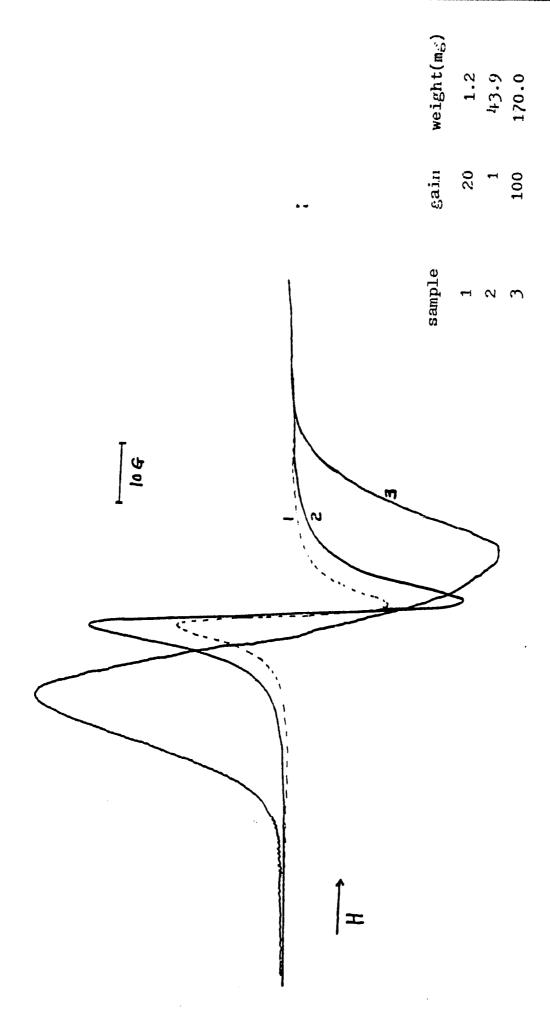
as cured, 2- 100 Mrad, 3- 1,000 Mrad. The sample was irradiated with electron beam and the doses indicated are cumulative ones. (1720 cm^{-1}) with irradiation dose Growth of carbonyl group Fig. 15.



ENEBGY

SURFACE

Figure 16. Plot of surface energy against irradiation dose of graphite fiber.



Superposition of ESR spectra for : 1-graphite fiber(as-received, 2- composite (as-received), 3- TGDDM-DDS epoxy (3 months after electron-irradiation, 5,000 Mrad). Fig. 17.

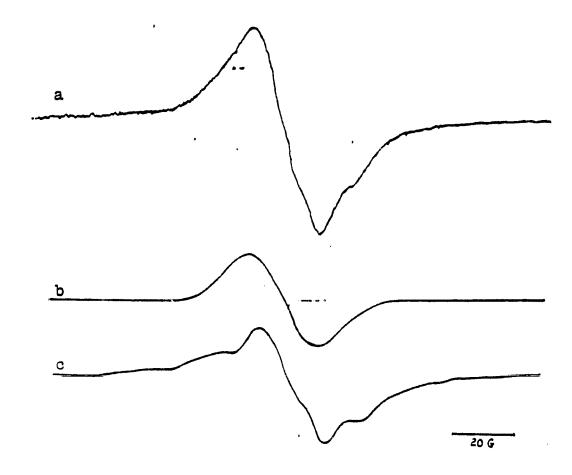
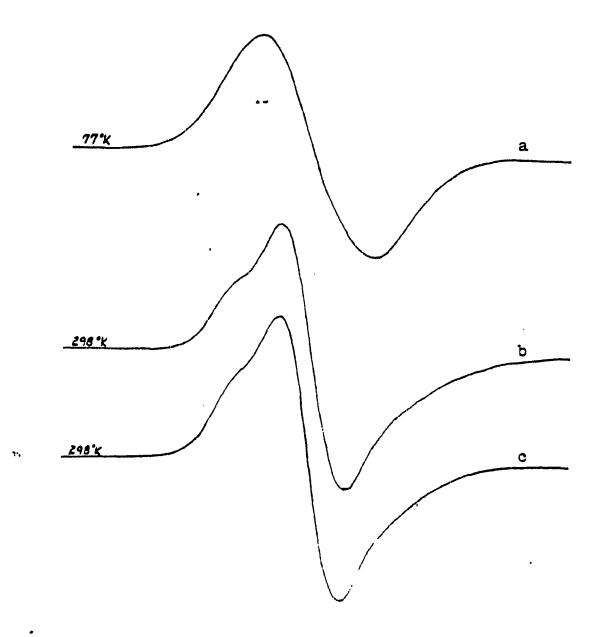


Fig. 18. ESR spectra of TGDDM-DDS epoxy irradiated with Y-radiation with a dose of 5 Mrad at liquid nitrogen temperature (-196°C): a. asirridiated, b. after 200 min exposure to air at 25°C, c. spectra obtained by subtracting spectra b from spectra a.



ESR spectra of epoxy irradiated at various temperatures.

- a- 77°K, %-radiation, 100 Mrad, 30 min. after irradiation, gain 1,
- mod. width 5.
 b- 298 K, Y-radiation, 100 Mrad, 30 min. after irradiation gain 5, mod. width 5.
 c- 298 K, electron beam (0.5 MeV), 160 Mrad, 5 min. after irradiation
- gain 1, mod. width 5.

Fig. 20.

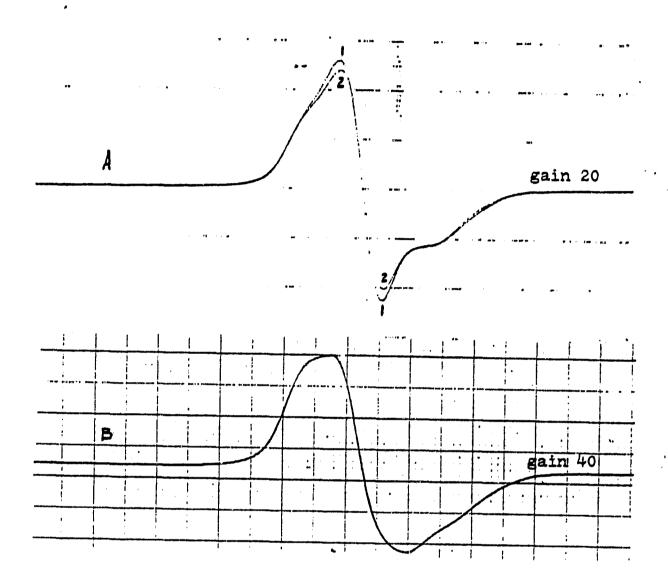


Fig. 21. ESR spectra of epoxy, Y-irradiated at room temperature with 800 Mrad: Al.as-irradiated in a vacuum-sealed glass tube, A2. as-irradiated in air, B. spectrum Al after 90 day exposure to air at 25°C.

VIII. Appendices

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2.4

Appendix A

$$\mathcal{T}_{LA} = \mathcal{T}_{LA}^{d} + \mathcal{T}_{LA}^{P} = \mathcal{A}_{L}^{2} + \beta_{L}^{2}$$

$$\mathcal{T}_{EA} = \mathcal{T}_{EA}^{d} + \mathcal{T}_{EA}^{P} = \mathcal{A}_{E}^{2} + \beta_{E}^{2}$$

$$W_a = V_{LR} (1 + COS\theta)$$

$$W_a = 2[\alpha_L \alpha_E + \beta_L \beta_E]$$
$$= W_a^b + W_a^b$$

$$\frac{W_a}{2\alpha_L} = \alpha_E + \beta_E (\beta_L/\alpha_L)$$

★ LA = SURFACE ENERGY OF LIQUID

X d = DISPERSION COMPONENT OF SURFACE ENERGY OF LIQUID

X EA. = SURFACE ENERGY OF EPOXY

X d = DISPERSION COMPONENT OF SURFACE ENERGY OF EPOXY

X P = POLAR COMPONENT OF SURFACE ENERGY OF EPOXY

 $\beta_L = (Y_{LA}^p)^{1/2}$

 $\beta_E = (\chi_{EA}^p)^{1/2}$

W = WORK OF ADHESION

Appendix B

Publications, Presentations and Theses Completed

Publications:

- 1. R. E. Fornes, J. D. Memory and N. Naranong, "Effect of 1.33 MeV γ Radiation and 0.5 MeV Electrons on the Mechanical Properties of Graphite Fiber Reinforced Composites," J. Appl. Polym. Sci. 26: 2061-2067 (1981).
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- 13. M. Kent, K. Wolf, J. D. Memory, R. E. Fornes and R. D. Gilbert, "The Effect of 0.5 Mev Electron Radiation on the Crystallinity of Carbon Fibers in Composites," <u>Carbon</u>, <u>22</u>: 103-104 (1984).
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Presentations:

R. E. Fornes, J. D. Memory and R. D. Gilbert, "Effects of Ionizing Radiation on the Mechanical and Structural Properties of Graphite Fiber Reinforced Composites," Gordon Research Conference on Fiber Science, July 1981.

Seminars presented on this work at Georgia Institute of Technology (May 1981), at the Polymer Poup meeting, North Carolina Division, American Chemical Society, Raleigh, NC (January 1981), at the Jet Propulsion lab (1983, three seminars), at Lawrence Livermoor Labs (1983, two seminars), at NASA Langley (1981, 1982, 1983) and at the U. Tennessee (1984), Fiber-Polymer Science Seminar at NCSU (1984), Nuclear Engineering Seminar at NCSU (1984), Honors Seminar at NCSU (1984) (see Bull. Amer. Phys. Soc. citations listed above).

Theses completed:

- 1. Naraporn Naranong, M.S. Thesis, "Effect of High Energy Radiation on Mechanical Properties of Graphite Reinforced Composites," (1980).
- Kevin Schaffer, M.S. Thesis, "Characterization of a Cured Epoxy Resin Exposed to High Energy Radiation with Electron Spin Resonance," (1981).
- 3. George M. Kent, M.S. Thesis, "X-ray Diffraction and ESR Studies of the Effects of High Energy Radiation on Composite Materials," (1982).
- 4. Kay W. Wolf, Ph.D. Dissertation, "Effect of Ionizing Radiation on the Mechanical and Structural Properties of Graphite Fiber Reinforced Composites," (1982).